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## Contribution of a solute atoms in the relaxation phenomenon at high temperature in Cu-Al single crystal alloys

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### Abstract

Two Cu-Al single crystals with 7 and 14 at. % Al respectively have been studied using isothermal mechanical spectroscopy (IMS) technique. After a 1% cold work by torsion, the samples have been progressively heated to 1140 K and then cooled until room temperature. IMS experiments allow to compare the isothermal internal friction spectra obtained during the heating (in this case, the annealing temperature is equal to the temperature of measurement) with the measurements performed at various temperature during the cooling after the annealing at 1140 K.

Three relaxation peaks were observed. The first one at about  $0.4 T_M$  ( $T_M$ : melting point) is a Zener relaxation peak ( $P_Z$ ) due to the reorientation under constraint of pairs of aluminium atoms. The high temperature annealing does not influence  $P_Z$ .

At about  $0.6 T_M$ , a peak ( $P_1$ ) related to a dislocation mechanism is evidenced. The relaxation strength of  $P_1$  peak decreases with the temperature and a new relaxation peak ( $P_2$ ) is progressively developed. The IMS spectra obtained during the cooling evidenced only  $P_2$ .

The relaxation parameters obtained by the Arrhenius plots and the evolution with the annealing temperature allow to assign  $P_1$  and  $P_2$  to an interaction between the dislocations and the solute atoms according to the Darinskiy model.

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### 1. Introduction

The monophases alloys are of particular interest to the study of relaxation phenomena related to interaction mechanisms: dislocations-solute atoms. The sensitivity of the stacking fault energy  $\gamma$  to the concentration of solute atoms and their solubility preferential in the fault (Suzuki effect) make the study of the Cu-Al alloys interesting to highlight the influence of this parameter. In fact, the stacking fault energy of these alloys strongly depends on the aluminum at least for contents in the range 0-19 at. %. This parameter, which controls the width of decoupling partials, plays an important role in the nature of sliding dislocations. This work reports the results of experiments

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obtained by IMS on two single crystals of Cu-Al alloy (7 and 14 at%) during heating after 1% cold work followed by an in-situ annealing at 1140 K. The presented results are focused on two relaxation peaks observed on the tow samples in the temperature range 750 K-1140K. Their presence in single crystals of pure metals evidenced by different authors [1-9-10-11], has allowed isolating the relaxation phenomena associated to the interaction: dislocations - solute atoms. These peaks appear to be consistent with those provided by the Darinskiy model. [2]

## 2. Experimental procedure

Isothermal internal friction experiments were carried out with a forced torsional pendulum under a vacuum of  $10^{-8}$  Pa. The pendulum and the experimental technique have previously been described [3]. In the case of the forced vibrations,  $Q^{-1}$  is equal to  $\tan\phi$  where  $\phi$  is the phase lag between the applied stress and the resulting strain. The measurement frequency ranged between 10 Hz and  $10^4$  Hz and five frequencies per decade were used. Experiments were performed with a maximum strain amplitude of  $10^{-5}$ . The CuAl single crystals samples were cut by spark machining in bars of dimensions: 40 mm  $\times$  4 mm  $\times$  1 mm. Then the bar was chemically polished to remove the volume affected by machining. 1% cold worked in torsion. X ray diagrams made before and after internal friction experiments showed that no recrystallization occurred in the samples during the high temperature tests.

## 3. Experimental results

We will use these abbreviations for all the experimental results:

$T_M$  = measurement temperature,  $T_A$  = annealing temperature,  $T_m$  = melting temperature.

### 3.1. Cu-Al (at.7%)

The internal friction spectra obtained during the first heating after the 1% cold work exhibit a relaxation peak as shown in Figure 1. In the experiments at the temperatures rose successively from 825 to 963 K, this peak ( $P_1$ ) decreases and shifts towards higher frequencies as the annealing temperature increased. The increase of the relaxation strength for temperatures higher than 1023 K indicates that a new peak ( $P_2$ ) grows progressively. After the subtraction of the low frequency background, figure 2 shows that the  $P_1$  peak disappears in aid of  $P_2$ . The subtraction method of the background has been described in detail elsewhere [12]. After annealing at 1140 K, the internal friction spectra display only  $P_2$  peak down to low temperature (Fig.3), its height decreases with the temperature of measurement. Fig. 4 shows the Arrhenius plots of the Napierian logarithm of the peak frequencies versus the inverse temperature. It turned out that they are not linear for the two peaks at the first heating. On the contrary, after annealing at 1140 K, the plots are straight lines and the corresponding apparent activation parameters are: limit relaxation time  $\tau_0 = 2 \times 10^{-9}$  s and activation energy  $H = 188$  kJ/mol, at 1Hz the peak maximum would be located at about  $0,9 T_m$ . These curves show a relatively stable microstructure until about 1023 K then a transition in which the existing microstructure evolves to another more stable at highest temperature. These results show that the annealing at 1023K remains insufficient to obtain a stable configuration of high temperature, which was obtained only after an annealing at 1137 K. The influence of annealing appears clearly on  $P_1$ , it collapses in favour of  $P_2$  (Fig. 5). Figure 6 shows the influence of the annealing temperature on  $P_2$ .

### 3.2. Cu-Al (at.14%)

The appearance of the internal friction spectrum of this sample during the first heating remains the same as the previous alloy. Figures 7 and 8 show respectively the relaxation spectra of  $P_1$  and  $P_2$  peaks during the first heating. The height of  $P_1$  decreases with the temperature of measurement.  $P_2$  peak appears at 891 K superimposed at a low frequency background, its height increases with the annealing temperature. The Arrhenius curves of the tow states annealed respectively at 1093K and 1110K are plotted in figure 9. The plots for the heating after annealing at 1093K and those for the experiments performed upon cooling from 1110 K are parallel. This shows that the mechanism responsible for relaxation of high temperature remains the same. This result is confirmed by Figure 10 which represents the peak  $P_2$  at 1060K for the tow annealing temperatures (1093 and 1110K). The relaxation parameters have been deduced from these Arrhenius plots: the limit relaxation time  $\tau_0 = 1,11 \times 10^{-10}$  s and the apparent energy of activation  $H = 184,7$  kJ/mol. at 1Hz the peak would be located at about  $0,8 T_m$ .

### 3.3. Comparison between the two Aluminium contents

Figures 11 and 12 show respectively that the amplitude of the two peaks  $P_1$  and  $P_2$  is significantly higher in the lower aluminium content.

At high temperatures, figure 8 show that the relaxation peak  $P_2$  for the 14% aluminium content is superimposed at a high low frequency background.

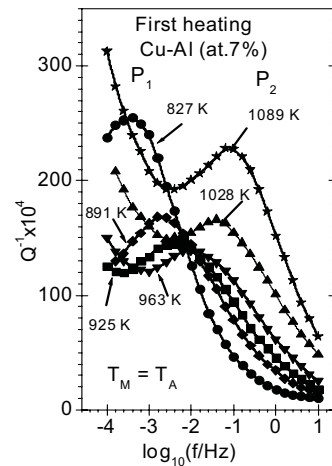


Fig. 1. Internal friction spectra obtained at various temperatures during the first heating after 1% cold work at room temperature.

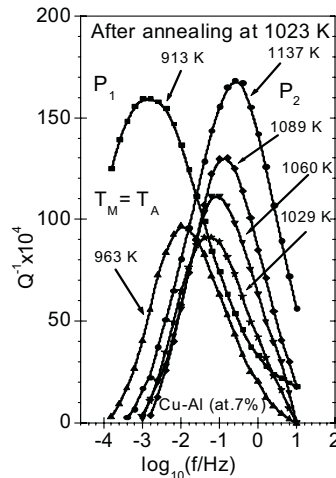


Fig. 2. Internal friction spectra obtained at various temperatures during heating after an annealing at 1023K

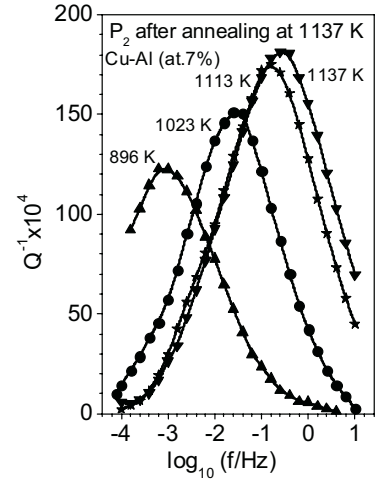


Fig. 3. Evolution of  $P_2$  peak for various temperatures during successive cooling after in-situ annealing at 1137 K.

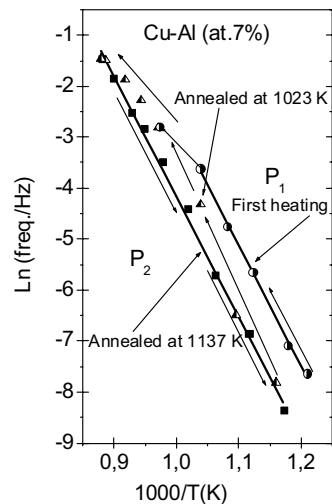


Fig. 4. Arrhenius plots of  $P_1$  and  $P_2$  peaks obtained during the first heating and of  $P_2$  peak after in-situ annealing at 1140 K.

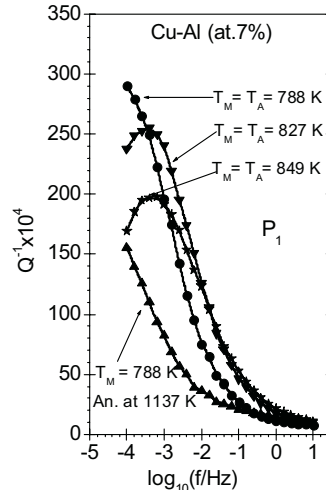


Fig. 5. Influence of the annealing temperature on  $P_1$ . This peak completely disappeared at 788 K after annealing at 1137 K.

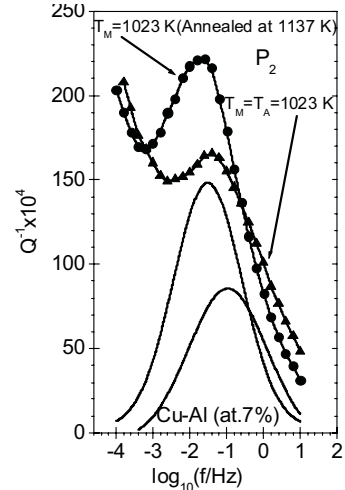


Fig. 6. The  $P_2$  peak becomes more pronounced and shifted towards lower frequencies with increasing annealing temperature.

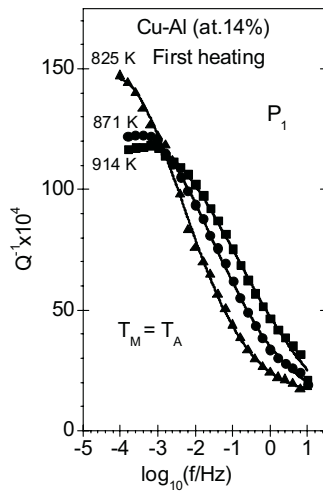


Fig. 7. Evolution of  $P_1$  peak for various temperatures during the first heating

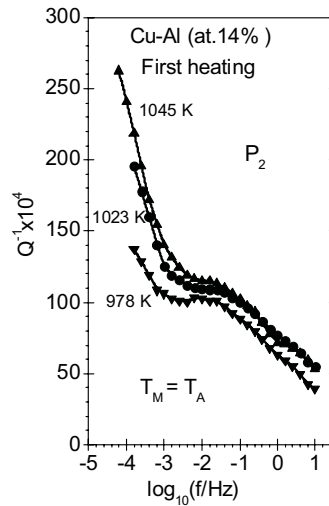


Fig. 8. Internal friction spectra obtained at high temperatures during the first heating.

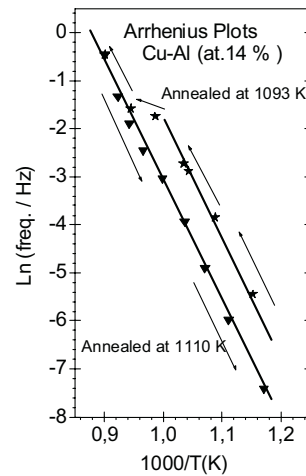


Fig. 9. Arrhenius plots obtained during heating after annealing at 1093 K and by cooling after annealing at 1110 K

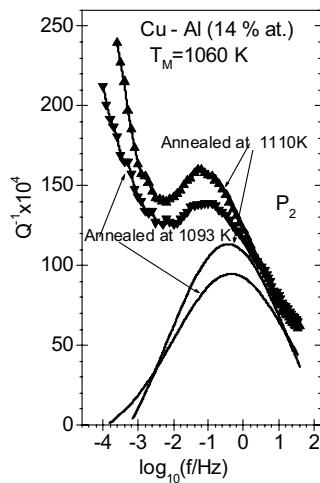


Fig. 10. Influence of annealing temperature on  $P_2$ .

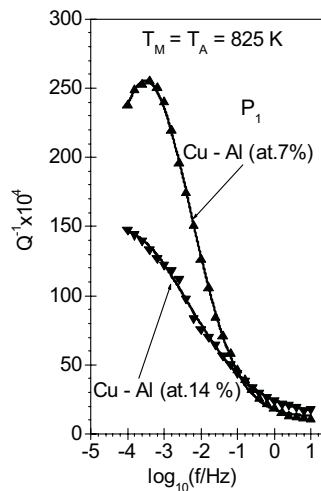


Fig. 11. Evolution of  $P_1$  at 825 K for the two samples

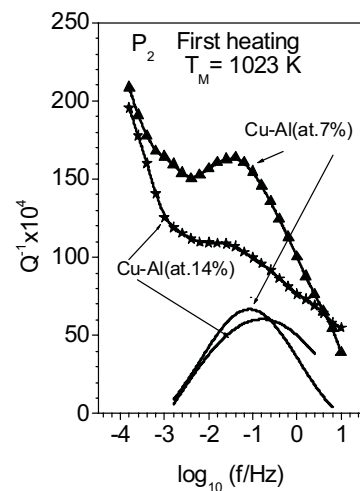


Fig. 12. Evolution of  $P_2$  at 1023 K for the two samples

#### 4. Discussion and conclusion

As we saw previously; the origin of these two peaks is closely dependent on the presence of dislocations and thus on their configuration.

The model of Darinskiy gives an account of these experimental facts since it also envisages two peaks respectively at low and high temperature:

For the low temperature peak: 
$$Q_{LTP}^{-1} = \frac{2\pi}{3} \frac{1-\nu}{2+\nu} \rho d_0^2$$

For the high temperature peak: 
$$Q_{HTP}^{-1} = \frac{8\pi}{9} \frac{1-\nu}{4-5\nu} \frac{\rho l^2}{\ln\left(\frac{L}{r_0}\right)}$$

(with  $\nu$  Poisson's ratio,  $L$  length of the band of dislocation,  $r_0$  ray of the loop of dislocation,  $\rho$  Density of dislocations,  $l$  length of dislocation between two inking points and  $d_0$  the stability width of the stacking fault).

Our results show for the two contents that the P1 peak is totally removed and after an annealing at high temperature (1140 K). According to [4- 5- 6 - 7-11], its origin is due to the dipoles and multipoles introduced by work hardening and blocked in their primary slip plane.

This behavior is perfectly compatible with the peak envisaged by the model of Darinskiy since the destruction of the dipoles involves a reduction in their density  $\rho$ .

However the comparison of P<sub>1</sub> for the two contents shows that the amplitude of this one is more significant for the 7% than for the 14%, which is in contradiction with this model since this last envisages an increase in the amplitude of this peak with the content (a reduction in the stacking fault energy involves an increase in  $d_0$ ).

Thus, P<sub>1</sub> peak is attributed to the dislocation microstructure created by the 1% cold work. During the first heating this microstructure progressively changes and a new dislocation arrangement spreads out giving rise to the appearance of P<sub>2</sub> peak. The values obtained for the limit relaxation time respectively for the two contents (7% and 14%) indicate that the P<sub>2</sub> peak is due to the movement of long segments of dislocation coming from the destruction of dipoles by recombination of partials [8]

The amplitude of P<sub>2</sub> is, in this case also, more significant in the 7% than in the 14%, which could be explained by the fact why the segments of dislocations present in the first content are longer ( $\tau_0$  of the 7% slightly higher than that of the 14%). That appears perfectly plausible in measurement or a greater content of solute atoms decreases the distance between the inking points and thus the length of the segments of dislocations.

This peak is, therefore, in perfect agreement with the peak P<sub>HT</sub> envisaged by the model of Darinskiy, since the dominating term in the relation of this last is:  $l^2$  ( $l$  = length of dislocations).

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